

PDI/ACE/048/98

Aim Preparation of ACE from 7-Ac  
(Bio-chem.)

"Pre-demonstration"

Repeation of PDI/ACE/047/98

STAGE-I

Prep<sup>n</sup> of TEA

2-Furyl chloride : 55.0 gm

NaSH : 60.0 gm

DMSO :  $\frac{1}{2}(730 + 950)$  ml

EtoAc :  $(500 + 200)$  ml

1:1HCl :  $(70 + 100)$  ml

NaHCO<sub>3</sub> : 40.0 gm

STAGE-II

Prep<sup>n</sup> of ACE

7-Ac : 100.0 gm

EtoAc : 400 ml

BF<sub>3</sub> : 137.0 gm

EDTA :  $(2.0 + 0.4)$  gm

TEA : 250.0 ml

SME : 2.0 gm/2

DMSO : 300.0 ml/2

DMSO

$\frac{1}{2}(200 + 100)$  ml

EtoAc

$400 + (50 \times 100)$  ml

B

12V NH<sub>3</sub> 8M

14 ml

10% NaOH

18.3 ml

DMSO

$(200 + 50)$  ml

$200 + 50$  ml

EtoAc

150 + 50 ml.

$(150 + 50)$  ml

23

Kit/vid. 8 013 242.4 / 250ml 95.25%

Kit/vid. 8 Alz 478.3 / 450ml 90.09%

R/M reporting 2016

1 hr 51 min 14.35% 11.02% 83.09% loop

240 hrs 100.34% 7.77% 11.89.53% a92%

Day 2016 56.7 58.9

MIC 100% 88.7

227

PDL/ACF/05/198

Ami Preparation of ACE from 7-Aza (Bio chemi)

Raw materials

Stage-I, Prep<sup>n</sup> of TFA

2-Furyl chloride	: 55.0 gm
NASH	: 60.0 gm
DMSO	(700 + 30 + 350) ml
Etmer	(500 + 200) ml
1:1HCl	(70 + 10) ml
NaHCO <sub>3</sub>	" 40.0 gm

: 93.7

Vd. of TFA : 260.0 ml

Stage-II, Prep<sup>n</sup> of ACE

TACA	: 100.0 gm
EbAc	: 400.0 ml
BF <sub>3</sub>	: 137.0 gm
TEA	: 26.0 ml
EDTA	: 0.5 + 0.5 gm
SNS	: 2.0 gm
DMSO	: 800.0 ml

: 132. NH<sub>3</sub> 82.7

: TfOAc : 100 + 300 + 600

: Rec. Reci. DILW : 300 + (dx 10) ml

wef wt: 2.55. F<sub>2</sub> =

PDL/ ACF/ 053/90.

Aim:- Preparation of AACF from 7-ACA (Biochem)  
 Modif: 130 gms  $\text{BF}_3$  gas purged instead of 137 g.

Raw materials Stage - I

$\text{Eu}(\text{Ph}_3)_3$  : 55.0 gm

$\text{NaBH}_4$  : 60.0 gm

DMSO : (730 + 50) ml

EthAc : (500 + 200) ml

THF : (70 + 100) ml

$\text{NaHCO}_3$  : 40.0 gm

Stage - II

7-ACA : 100.0 gm

EtAc : 400. ml

$\text{BF}_3$  : 130.0 gm

TFA : 80 ml

SHS : 2.0 gm

EDTA : (2.0 + 0.5) gm

DMSO : 300 ml

7-ACA : 200.0 ml pH 3.65

EthAc : (100 + 300 + 100) ml

DMSO : (300 + 100 + 100) ml

No. Rec. Non-toxic

Terminated to 360.

Actual wt. of 69.0 gms.

Actual DMSO : 550 ml

Actual TFA : 3.25 ml

used in PDL/ ACF/ 053/90 & 054/90

PDL/ACE/055/98

AIM: Preparation of ACE from FACA (Biochem)  
by

Raw materials Stage-I TFA prep?

2-Furyl chloride : 55.0 gm

NASH : 60.0 gm

DMSO : (500 + 30 + 50) ml

EtoAc : (500 + 200) ml

1% HCl : (723 + 100) ml

NaHCO<sub>3</sub> : 40.0 gm

Stage-II

ACE prep?

FACA : 100.0 gm

EtoAc : 400.0 ml

BF<sub>3</sub> : 137.0 gm

EDTA : (200 + 6) gm

TEA : 25.0 ml (99.68%)

SH<sub>3</sub> : 2.0 gm

13% NH<sub>3</sub> soln : 200.0 ml

DMSO : 300.0 ml

EtoAc : (100 + 300 + 100) ml

DMSO : (300 + 100 + 100) ml

R/m report: FACA ACE TFA top

Initial : 0.35% 85.61% 8.16% 3.42%

1 hr : 0.35% 85.50% 8.26% 3.67%

Terminated in 3 hrs

Wet wt : 249.7 gm

Aim: Preparation of ACE from 7-ACA.

Raw materials:

Stage-I prep<sup>n</sup>g TEA

2-Furyl chloride	55.0 gm
NaOH	60.0 gm
DMSO	(700+30+350) ml
Ethac	(500+200) ml
H <sub>2</sub> HCl	(75+100) ml
NaHCO <sub>3</sub>	40.0 gm

Stage-II prep<sup>n</sup>g ACE

TEA : 255.0 ml (sys problem)

7-ACA	100.0 gm
Ethac	600.0 ml
BF <sub>3</sub>	127.0 gm
EDTA	(9.0+0.6) gm
SHS	2.0 gm
DMSO	300.0 ml
13x NH <sub>3</sub> sol.	240 ml
Ethac	(100+300+100) ml
DMSO	(300+100+100) ml

Run repeat: 7-ACA ACE TEA time

2 hrs	8.32	82.74	6.62	3.50
3 hrs.	0.31	88.09	6.27	3.93

Terminated in 3 hrs.

Wet wt: 276.4 g.

Aim: Preparation of ACF from TAc.

Raw materials

Stage-I prep'g TEA

2-Euroyl chloride	: 55.0gm
NaSH	: 1.60.0gm
DMW	: (100+30+30)ml
EtoAc	: (500+200)ml
H <sub>2</sub> O	: (75+100)ml
NaHCO <sub>3</sub>	: 40.0gm

prep'g ACF Stage-II

TAc	: 100.0gm
EtoAc	: 400.0ml
BF <sub>3</sub>	: 137.0gm
EDTA	: (2.0+0.6)gm
TEA	: 255.0ml
STS	: 20.8gm
DMW	: 200ml
13% NH <sub>3</sub> Soln.	: 233ml.
EtoAc	: (100+300+100)ml
DMW	: (300+100+100)ml

R/m report TAc ACF TEA Imp.

2Yrs hrs 0.55 87.38 7.40 1.98

3 hrs 0.47 88.78 6.92 2.08

Terminated in 3 hrs.

Net wt : 226.3 gms.

Used in P.DI CFCI 157/98